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# Materials Research Department annual report 2002

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# Materials Research Department

## Annual Report 2002

Risø National Laboratory

May 2003

Risø-R-1404 (EN)

# 2002 - A Year of Growth and Expansion

Materials research is a key driver for the development of new products and devices. In the Materials Research Department we span areas from strategic long-term basic research all the way to technological development in collaboration with industry. Our focus is on materials for new energy technologies within the production, storage, transport and conservation of energy, but our research also has spin offs in many other fields and applications. Our aim is to perform research of the highest international quality, but also research that is of direct relevance and benefit to society.

The Department grew significantly larger in 2002 with the recruitment of many new employees during the year, especially within our solid oxide fuel cell research programme. The Department has about 10% more staff than a year ago. This is of course a very positive sign, but has lead to some difficulties with office and laboratory space. In the coming years we plan to expand even further, in terms of students, especially at the Masters project level.

One important event was the transfer of the old physics department – from where it had been located for more than 40 years - into the materials research buildings on the 'island', so that nearly all the staff are now located on a common 'campus'.

Another significant event was the expiration at the end of 2002 of the Engineering Science Centre, which had been led successfully by Torben Leffers for 10 years. During its existence the Centre was a major driving force in the initiation of many new projects that today have developed into major activities in their own right.

From the first of January 2003 the Department was given a new organizational structure with five new research programmes that provide a better match to the changing conditions in the Danish research system. Although the annual report covers 2002, we felt it natural that the annual report should be organized in themes that reflect the new structure.

There have been many scientific highlights in 2002. We have chosen a few to be presented in the annual report to give you a flavour. Furthermore, a number of important milestones were also met in 2002; achievements that are particularly important for the scientific development of the Department.

*The Department has come a long way.*

### The inauguration of the pre-pilot plant for manufacturing solid oxide fuel cells

This facility is one of the corner stones in our fuel cell activities. The pre-pilot plant enables us to manufacture large numbers of cells for the production of cell stacks, and facilitates cell lifetime tests in order to mature the technology.

### The inauguration of the new high resolution Jeol transmission electron microscope

The new transmission electron microscope gives direct insight into the atomic scale of materials and will enable the Department to be at the forefront in nanoscience and nanotechnology.

### The inauguration of the small-angle-neutron-scattering instrument (SANS)

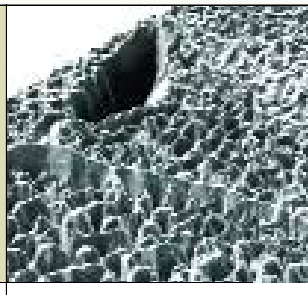
The SANS instrument has been transferred from Risø's DR3 reactor to the Paul Scherrer Institute in Switzerland. The instrument underpins Risø's role as a national front runner in the exploitation of large-scale facilities for materials science.

We are also very pleased that in 2002 prizes and awards honoured employees on the staff. Larry Margulies received the Young Scientist prize at the European Synchrotron Radiation Facility ESRF for his significant contribution to establishing the 3-dimensional x-ray microscope. Karin Vels Hansen received the A.R. Angelo prize for her interface studies on electrodes in solid oxide fuel cells. Bachu Singh was appointed to be a member of the British EPSRC Peer Review College, and Morten Eldrup was appointed Adjunct Professor in positron chemistry at Lund University.

*Robert Feidenhans'l*

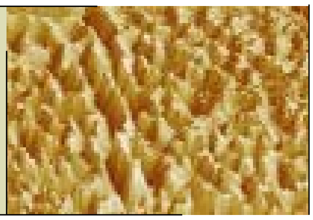
## Staff

The Department comprises about 155 people, including many students. Out of these, 50 are permanent scientific staff, including 2 research specialists and 4 research or adjunct professors. There are 30 positions as researchers and post docs on time limited contracts. This gives the Department a very flat age profile.



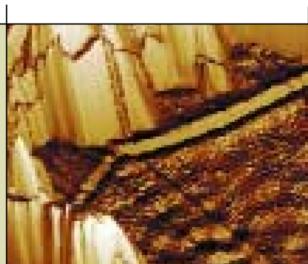
## Education

The Department is involved in a variety of educational activities. The Department had 27 Ph.D. students, of whom 4 completed their degrees in 2002, and 12 Masters students.



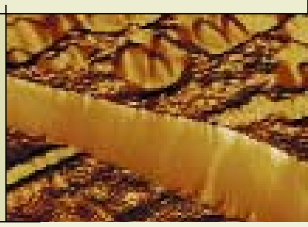
## Publications

The quality of research is measured by the publications. The Department has a good international standing with 114 papers in international journals, including several in the most prestigious journals; and many conference papers, books and reports.



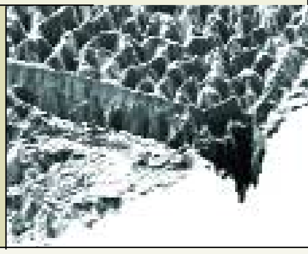
## Organizational activities

Several staff members are engaged in international and national organizational work for professional societies, conference arrangements, etc..



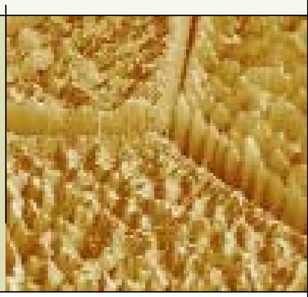
## Economy

The turnover of the Department is 116 Mkr. (15.6 M€) out of which 52 Mkr. (7M€) comes directly from Risø, 48 Mkr. (6.4 M€) from programme supported research and 16 Mkr. (2.2 M€) from companies and commercial income.



## Innovation

The Department has a major industrial technology transfer programme with Haldor Topsøe A/S for the development of solid oxide fuel cells. There is a major collaborative effort on materials developments and testing within the Danish windmill industry and many other collaborations on a smaller scale.

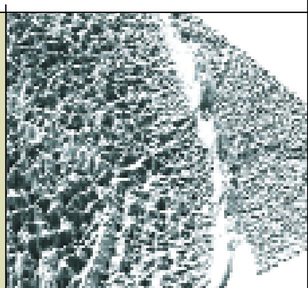


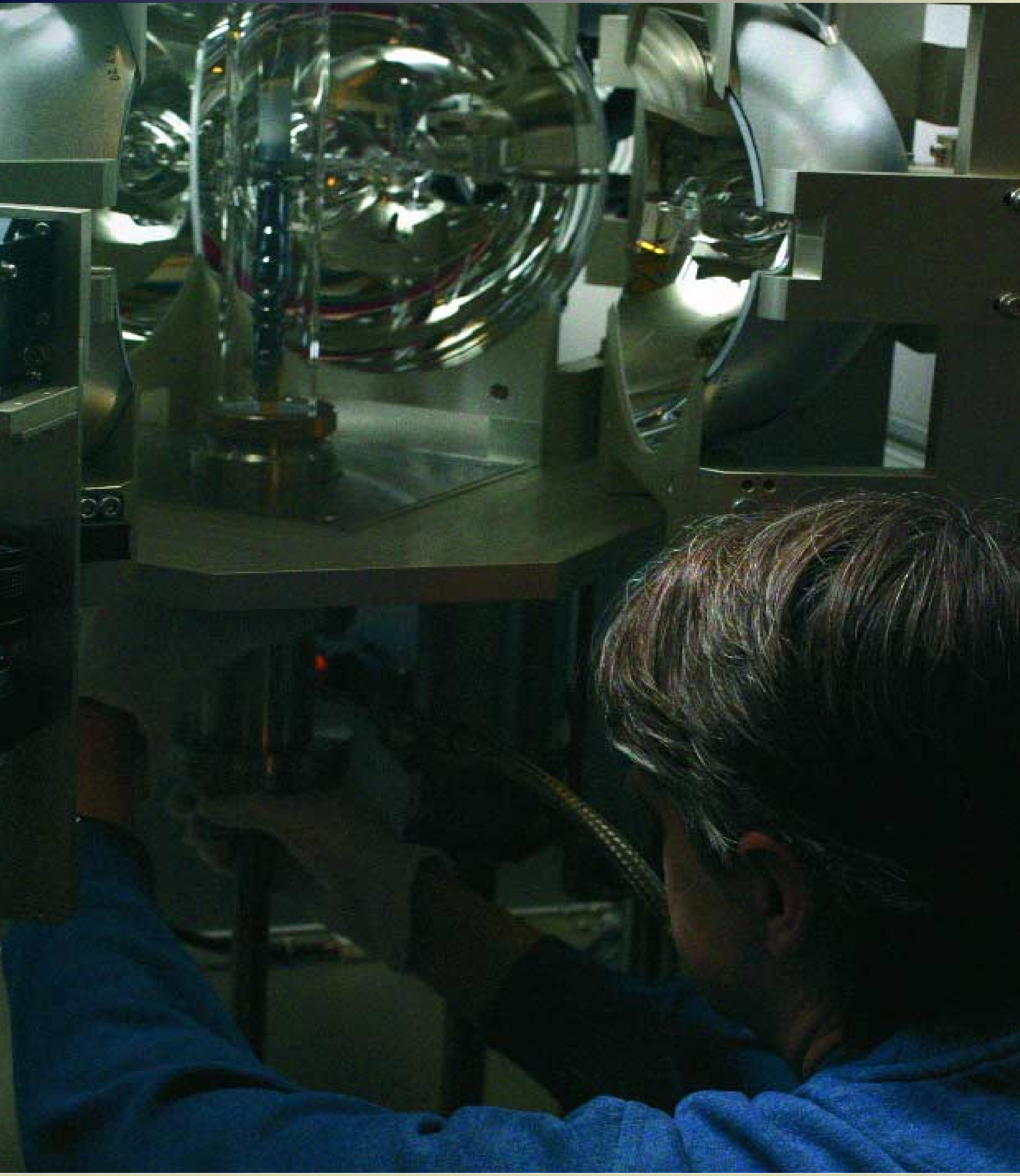
## Structure

As of 1st January 2003 the Department is organized into five programmes:
*Superconductivity and Magnetism* headed by Niels Hessel Andersen
*Fuel Cells and Materials Chemistry* headed by Søren Linderoth
*Nano- and Microstructures in Materials* headed by Allan Schrøder Pedersen
*Composites and Materials Mechanics* headed by Povl Brøndsted
*Metal Structures in Four Dimensions* headed by Dorte Juul Jensen

## Sponsors

We thank all our collaborators and sponsors for the support we have received in 2002, in particular the Danish Energy Agency, Elkraft System, Eltra, Haldor Topsøe A/S, the Danish Natural Science Research Council, the Danish Technical Research Council, the Danish National Research Foundation, Euklid, and the European Commission.





*View of the mirror furnace for crystal growth. The materials are kept inside a quartz tube to allow for the use of controlled atmosphere and protect the mirrors against evaporation from volatile elements during the growth process. Jean-Claude Grivel is adjusting the position of the seed crystal from below the furnace.*

# Superconductivity and Magnetism

The programme on superconductivity and magnetism comprises materials synthesis, studies of basic properties and strategic developments aiming at applications. Superconductivity and magnetism are strongly linked. Usually they are considered as mutually exclusive phases, but they are often formed by the same type of electronic pairs carrying opposite spins. Studies of magnetic properties are therefore key topics in the R&D activities. Applied magnetic fields are clearly detrimental to anti-parallel spin alignments and high fields may destroy superconductivity as well as antiferromagnetism. However, superconductivity may co-exist with antiferromagnetism and even with weak ferromagnetism, and it may survive in quite strong magnetic fields by confining the magnetic flux in tubes shielded by superconducting vortex currents.

The magnetic properties are essential for the general understanding of superconductivity, not least for the basic mechanism behind high-temperature superconductivity, and they are decisive for the technological development of new materials with higher current carrying capability. The strategic part of the programme includes development of superconducting tapes for power transmission, based on the high temperatures superconductor $Bi_2Sr_2Ca_2Cu_3O_{10}$, and for magnetic applications, e.g. in power generators for wind-turbines, by use of the newly discovered magnesium-diboride ($MgB_2$) superconductor. Key phenomena being studied in the interplay between magnetism and superconductivity include the role of magnetic fluctuations on the formation of the high-temperature superconducting state and the properties of the magnetic flux in the vortex tubes. The magnetic field in the vortices interact with transport currents and if insufficiently pinned they will move and cause losses that are detrimental for superconductivity and applications.

In magnetic materials basic studies are performed on nanomagnetism and quantum critical systems. Magnetism on the nanoscale includes novel phenomena as superparamagnetism, i.e. detachment of the magnetic moment from the crystal lattice due to thermal fluctuations, and enhanced collective oscillations of the spins. Investigating the mechanisms behind these quantum effects are essential for understanding and designing the magnetic devices of the future, e.g. for computers. Additionally, the interplay between magnetism and superconductivity is studied in nanoparticles comparable in size to the superconducting coherence length.

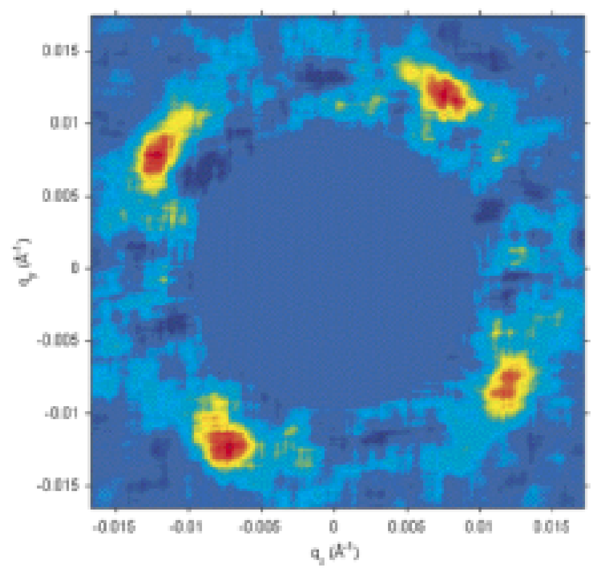
Neutron scattering is a primary technique for the experimental studies in the programme. During the past year the SANS (Small Angle Neutron Scattering) spectrometer from the DR3 reactor has been transferred successfully to the Paul Scherrer Institute (PSI) in Switzerland. Accordingly two of the three spectrometers included in the collaboration contract between PSI and Risø have been transferred on schedule. In year 2002 also the facilities for materials synthesis and for magnetic and transport characterisation have been moved to new environments. This completes the unification of programmes from the former Condensed Matter Physics and Chemistry Department with the Materials Research Department. The experimental facilities have been improved significantly during year 2002 by purchase of a mirror furnace for single crystal growth. This acquisition has been granted by the Technical Research Council via a national Framework Programme on superconductivity R&D.

*Niels Hessel Andersen*

## Flux Line Lattice in Superconductors

One of the characteristic properties of the High Temperature(HTc) superconductors is that they collect magnetic field in tube shaped regions in which the superconducting state is suppressed. These tubes are denoted flux lines and can order in a periodic lattice, which is called the flux line lattice. $La_{2-x}Sr_xCuO_4$ was among the first HTc superconductors discovered and recently a transition from a hexagonal flux line lattice below applied magnetic fields of H = 0.5 T and into a square symmetric lattice above H = 1.0 T was observed in a Small Angle Neutron Scattering (SANS) experiment. Neutrons possess a magnetic moment and are ideal for examinations of the flux line lattice in superconductors, because they can be scattered by the periodic magnetic flux structure. The figure shows the neutron diffraction pattern from the flux line lattice induced by an applied magnetic field along the c-axis of the orthorhombic unit cell. Flux lines repel each other and usually order in the close packed hexagonal lattice. However, the observation of a square flux line lattice could reflect the d-wave nature of the order parameter of the HTc superconductors or the fourfold symmetry of the Fermi surface of the conduction electrons. An alternative explanation is pinning of the flux lines to crystallographic defect structures called twin domain walls, which are formed, because the crystal structure changes from tetragonal at room temperature and into orthorhombic symmetry at low temperature. A pressure cell applying an uniaxial pressure of P = 4 MPa to the crystal was used in the SANS experiment to prevent the twin formation, but no effect of the de-twinning was seen. The flux line lattice was only observed in a x = 0.15 crystal, whereas a under-doped crystal with x = 0.10 showed no sign of the lattice. This is probably connected to a decomposition of the flux lines as superconductivity is confined in 2-dimensional Cu-O planes inside the unit cell when the doping is decreased. Future studies of over-doped crystals (x > 0.17) are planned, because the crystal structure remains tetragonal at low temperature and the superconducting phase has 3-dimensional character.

*Asger Abrahamsen and Niels Hessel Andersen*




*Diffraction pattern of neutrons scattered on the flux line lattice in the high temperature superconductor $La_{2-x}Sr_xCuO_4$ (x=0.15) induced by an applied field H||c = 1.0 T at T = 4.0 K. The fourfold symmetry of the pattern shows that the flux lines are ordered in a square lattice.*

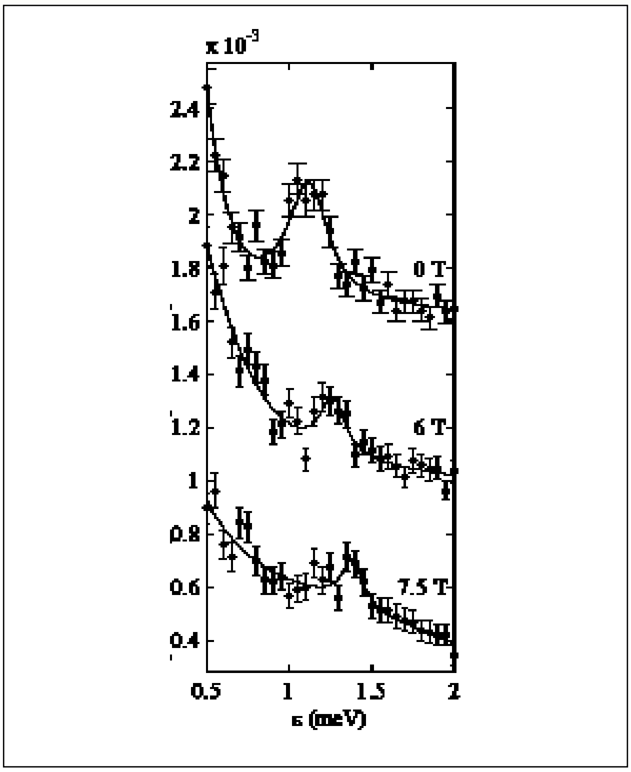
## Collective Magnetic Excitations in Nanoparticles

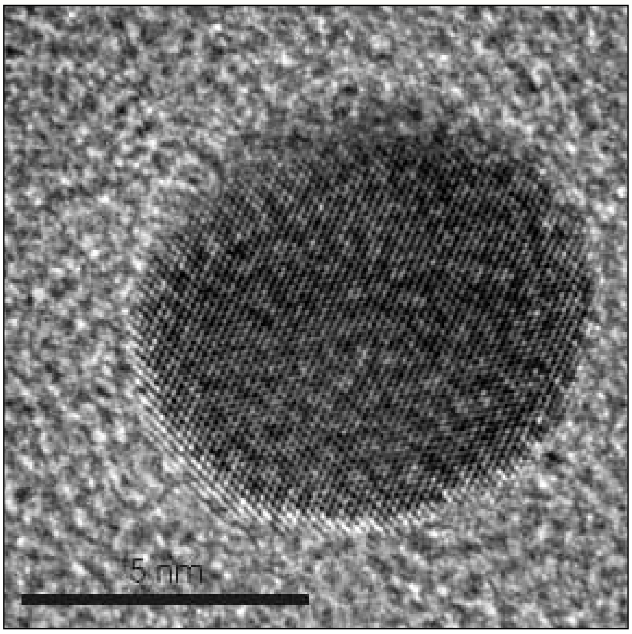
One of the major research topics of the Nanomagnetism group is spin dynamics in single domain magnetic nanoparticles. As the nanoparticle size decreases finite-size effects influence the spin dynamics and the quantum nature of the magnetic properties becomes evident. Reversal of the full nanoparticle magnetic moment induced by temperature, i.e. superparamagnetism, is one example. Another example is collective magnetic excitations, i.e. coherent precession of all the spins in a single nanoparticle. Understanding the mechanisms behind these effects is a challenge and crucial for designing new materials for magnetic components in computer technology.

Recently we have discovered a new collective magnetic excitation in nanoparticles of the iron oxide hematite, $\alpha$-$Fe_2O_3$. The nanoparticles were chemically produced at DTU. The spin dynamics was studied by inelastic and polarized neutron scattering at the Risø RITA-II and the TASP spectrometers at the Paul Scherrer Institute. The collective precession has a characteristic energy which is visible as a peak in the neutron scattering spectrum at $\varepsilon = 1.1$meV. Applying an external magnetic field speeds up the precession and narrows in the opening angle, which results in a movement of the peak to higher energies.

These findings are direct evidence for the discreteness of the collective magnetic excitations in nanoparticles, and future experiments are planned for measuring the predicted spin wave gap.

*Stine Nyborg Klausen, Luise Theil Kuhn,*
*Kim Lefmann and Per-Anker Lindgård*




*A transmission electron microscope picture of a single crystalline hematite nanoparticle placed on a thin carbon substrate. The graph shows the neutron scattering signal from the newly discovered mode of collective magnetic excitations and its behaviour with an externally applied magnetic field up to 7.5T.*

*Rasmus Barfod is inspecting the long term (>2000h) solid oxide fuel cell test stands.*

# Fuel Cells and Materials Chemistry

Fuel cell systems will be one of the future technologies for a more efficient and environmentally friendly conversion of chemical energy into electricity and heat. At the Materials Research Department R&D on Solid Oxide Fuel Cells (SOFC) has been carried out for more than 20 years, in collaboration with both universities, research centres and industrial partners. In 2002 a major step forward was taken by the start of a closer collaboration between Risø and Haldor Topsøe A/S on the development of SOFC. The effort now includes more than 50 people at Risø. The prime aim is that the many years of R&D at Risø National Laboratory will be transferred to the commercial environment and will bring SOFC systems to the consumers.

In 2002 the pre-pilot manufacturing facility for SOFC was inaugurated by Helge Sander, the Minister of Science, and Haldor Topsøe, the founder of Haldor Topsøe A/S. The pre-pilot facility is an R&D facility for the development of large-scale fabrication techniques – for the demonstration of the up-scalability and reproducibility of the technology developed at Risø. Cells produced at the pre-pilot facility are being tested at various places around the world. The electrical performance of the Risø SOFC is among the very best world wide – and their mechanical performance is unique with regard to strength and flexibility.

The more fundamental research has provided a deep insight into the interface, structural and morphological properties of the SOFC. This development has in large been supported by the Danish Energy Agency. In 2002 this resulted in the development of a cell with significantly better properties, both with regard to performance and production. This makes it possible to reduce the operation temperature from 850°C towards 700°C which is very important for the durability and the costs of the other components of the fuel cell system.

Education of young scientists and candidates within the area of fuel cells, materials chemistry and related areas is an important issue of the activities. We are proud that one of our young scientists, Karin Vels Hansen, received the A.R. Angelo award for her contribution to the understanding of the effect of impurities on the interfacial phases developing at the electrolyte-anode interface – an activity involving methods such as electron microscopy, surface analyses, and impedance spectroscopy as important characterisation tools.

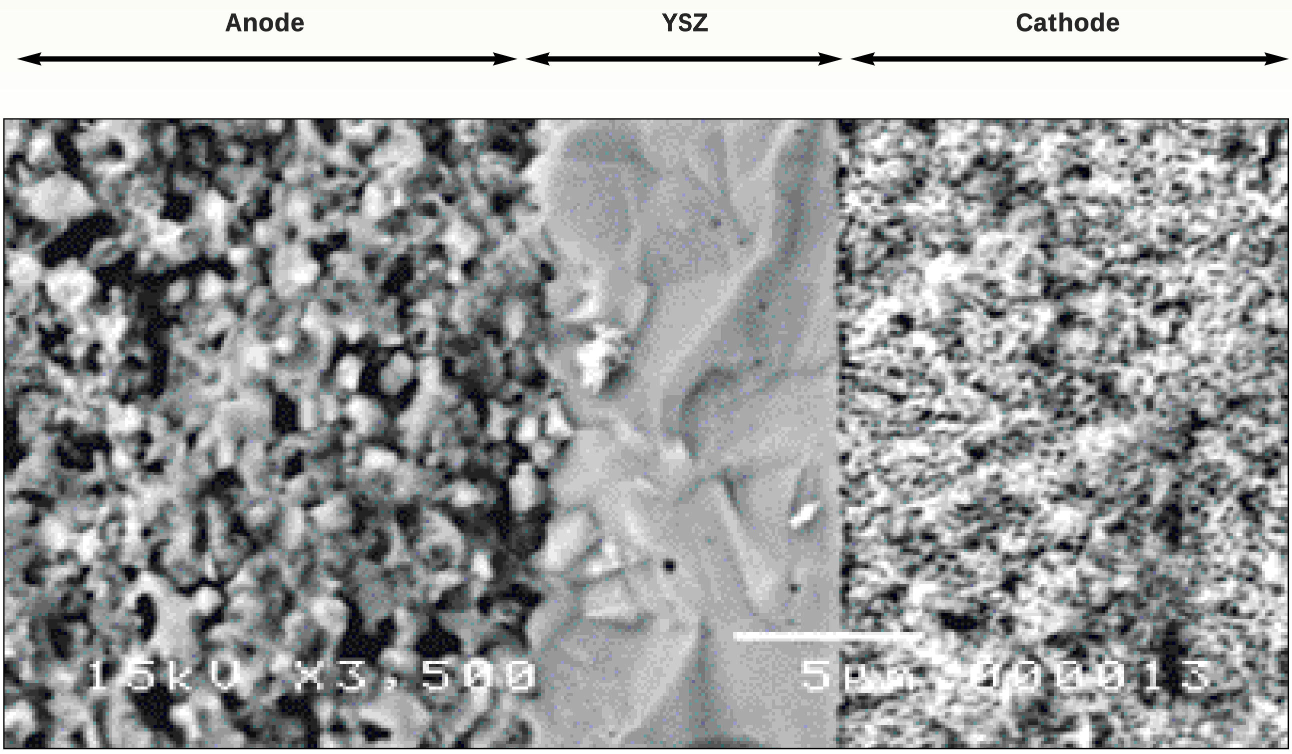
Students at universities are performing part of their final work in the Department, and the development of a course on fuel cells at the Technical University of Denmark, conducted by young scientists from Risø, was initiated in 2002. This effort will continue in the coming years.

*Søren Linderoth*

## Cathode development for SOFC

One of the key areas in current solid oxide fuel cell (SOFC) research and development is to reduce the operation temperature of the system, for example from 850°C to 700°C. This allows a significant cost reduction and improvement of the long-term stability of the SOFC system. Detailed analyses showed that the total cell loss at lower temperatures were dominated by the cathode losses. Cathode loss will contribute to the polarization resistance ($R_p$) and to the series resistance ($R_s$) due to resistance at the cathode/electrolyte interface. Chemical composition, cathode microstructure, as well as cathode/electrolyte interface structure were identified as the critical elements to be improved in order to reduce the losses. Investigation has been conducted in many aspects including stoichiometry of $(La_{1-x}Sr_x)_yMnO_{3\pm\delta}$ (LSM), noble metal doping, particle size distribution, and sintering profile. This effort has resulted in an $R_p$ as low as 0.06 $\Omega cm^2$ at 850°C obtained in our symmetric cell study and a low activation energy of 1.2 eV. Power densities of 1.44 W/cm$^2$ at 850°C and 0.5 W/cm$^2$ at 700°C have been achieved at a cell voltage of 0.7 V with an active area of 4×4 cm$^2$ in our anode supported cell. Processing optimisation has lead to a homogenous microstructure with sub-micron pores throughout the entire cathode and a good adhesion between cathode and electrolyte. This has resulted in a cell with area specific resistances of 0.175 $\Omega cm^2$ and 0.47 $\Omega cm^2$ at 850°C and 700°C, respectively. This is only half of our previous cell resistance. At the same time, the production time and the labour costs have been reduced significantly.

*Wei Guo Wang*

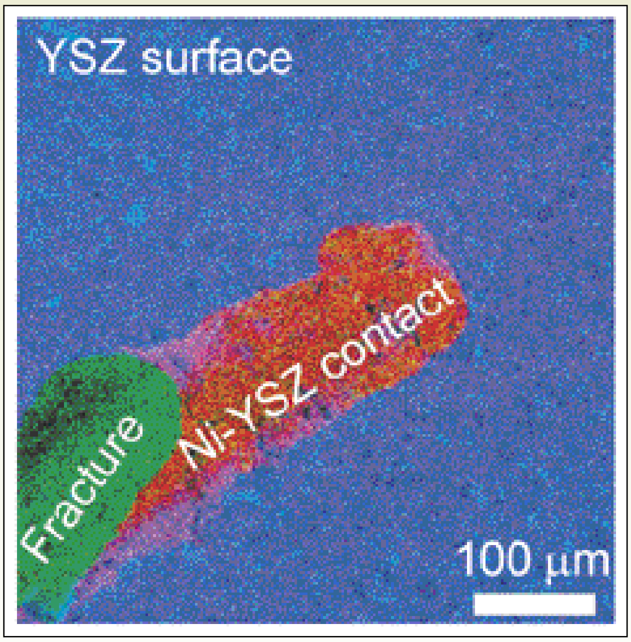



*SEM picture showing the improved microstructure of an anode supported solid oxide fuel cell (SOFC).*

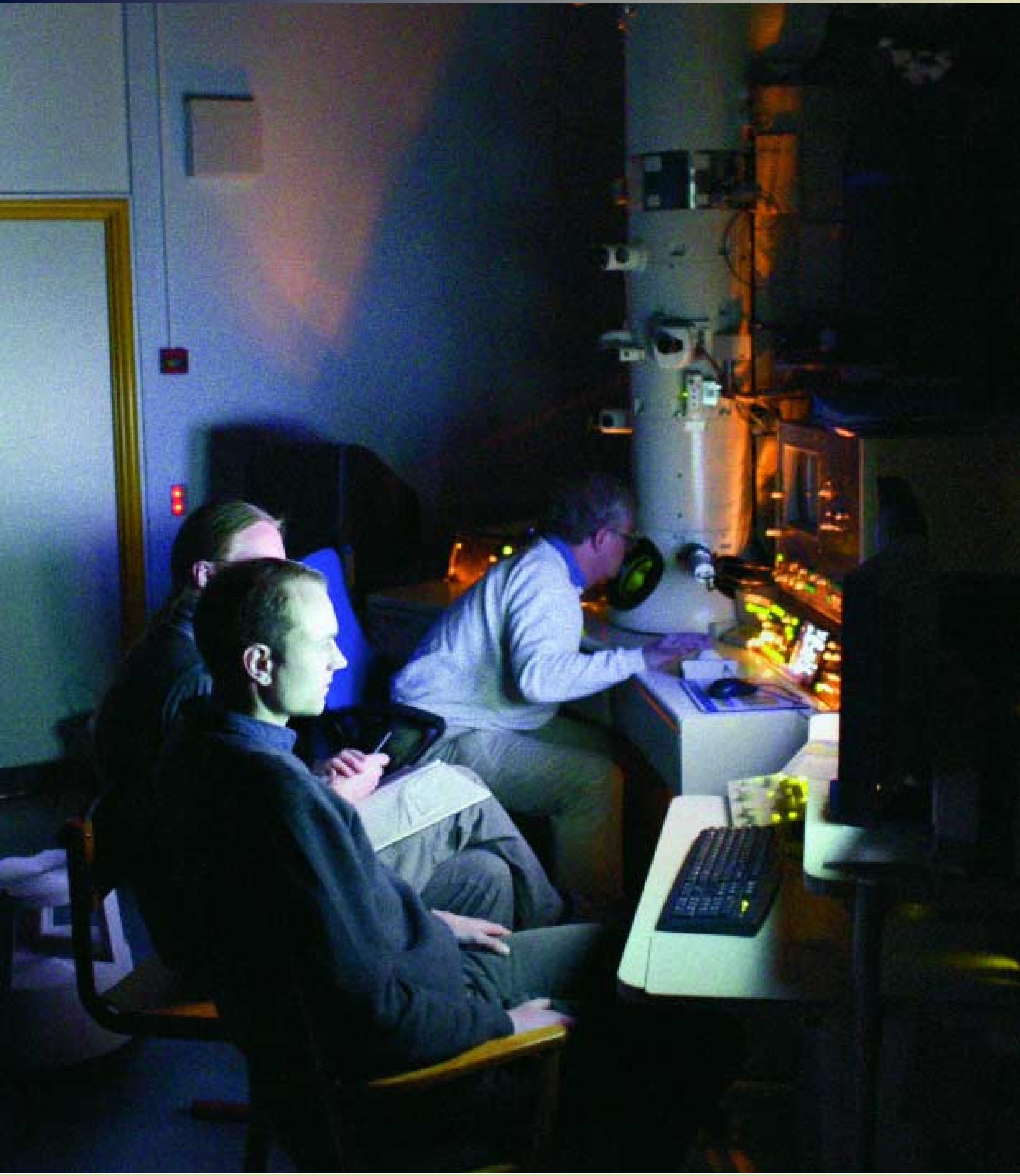
## The Ni/YSZ interface

The nature of the Ni/ yttria stabilised zirconia (YSZ) interface is important for the performance of solid oxide fuel cell (SOFC) anodes. In the present study, the Ni-YSZ interface has been examined with respect to changes in microstructure, chemical composition, and electrochemical properties. Two different types of nickel were used, an impure (99.8 %) Ni and a pure (99.995 %) Ni. The experiments were performed at 1000°C in a wet hydrogen atmosphere. The structural changes were studied with scanning electron microscopy and atomic force microscopy. It was found that the interface was dynamic, and that the impurities were affecting the morphology of the interface. The interface was examined with different advanced chemical analysis techniques: EDS, XPS and TOFSIMS, (in cooperation with the Danish Polymer Centre, Risø). It was found that the chemical composition of the interface was very dependent upon the impurity content of the Ni. Both the Ni and the YSZ contain small amounts of impurities, and elements such as Si, Al, Na, and Mn were found to segregate to the interface, and to the three-phase boundary between Ni, YSZ, and hydrogen, where the electrochemical reactions are assumed to take place. The electrochemical properties were examined with impedance spectroscopy, which showed a clear influence of the impurities on the performance of the anode. The area specific resistance for samples with a pure nickel anode was up to 10 times lower than for samples with an impure anode. Comparing the obtained data with those of the literature revealed that literature data correspond mostly to the impure anodes. It is believed that if the impurities can be controlled it may lead to a better performance of SOFC anodes.

*Karin Vels Hansen*




*Time of flight secondary ion mass spectrometry (TOFSIMS) image of a contact between impure Ni and yttria stabilised zirconia (YSZ). The red area is the actual contact. The colours imply that Mn and Na are present in the contact area. (Colour code: Mn red; Na blue; Y+Zr green).*



*Erik Johnson is using the new transmission electron microscope (JEOL 3000F) together with two scientists (Kristoffer Haldrup and Jesper Nygaard) from the University of Copenhagen.*

# Nano- and Microstructures in Materials

The Research Programme "Nano- and Microstructures in Materials" was formed by the fusion of the former three research programmes "Materials Models and Structure", "Radiation Damage, Defects and Fusion Materials" and "Powder Technological Materials". The aim has been to strengthen the activities within the overall Risø focus areas "Energy" and "Industrial Technology". It is now an important task for the newly formed programme in the future to maintain homogeneity in the work and to give strong and coherent contributions to the research work of the Department.

Two prominent events took place during 2002. One was the final commissioning of a brand new JEOL 3000F Transmission Electron Microscope. The other was the termination of a national Engineering Science Centre (IVC), which has reached many technical and scientific achievements during its 10 years operational period. This will be discussed separately on p.33. In addition the successful work done investigating the reaction of materials under conditions expected in future fusion reactors is given special attention on the following pages.

The Programme also comprises a number of other research projects. One of these concerns hydrogen energy. The interest in hydrogen as a vector in the energy supply system of developed countries has increased strongly in Europe during 2002. In an interview in October, the president of the European Commission expressed his expectations for the future use of hydrogen and likewise many member countries are initiating national funding for R&D in hydrogen for energy purposes. In the past Risø has worked intensively with pure magnesium for hydrogen storage applications. Work in this area has been strongly revitalised during 2002 by external funding from the Danish Research Councils and from the Nordic Energy Research Programme. It includes work on new alloys and other solid compounds for storage of hydrogen by direct solid gas reaction. The aim is to achieve capacities and reaction kinetics of interest for industrial applications, e.g. in the transport sector. A Ph.D. project has already been initiated and yet another one is planned to start during the year to come.

Another project deals with studies of bulk amorphous as well as partly crystalline alloys. Important findings about their mechanical properties have resulted. Bulk amorphous alloys are in the glassy state brittle when loaded in tension due to the lack of a work hardening mechanism. Nanocrystals embedded in an amorphous matrix may lead to an increase in fracture stress compared with the homogeneous amorphous alloy by suppression of shear band propagation. The effect has been studied in details for MgCuY bulk amorphous alloys. Controlled annealing for different lengths of time leads to partial crystallisation and in this way samples with various crystalline volume fractions were produced. The average size of homogeneously distributed $Mg_2Cu$ nanocrystals was about 100 nm. These partly nanocrystallized samples show an increase in yield or flow stress provided that the crystalline volume fraction is less than 50%. Larger volume fractions of nanocrystals promote brittle fracture of the specimen and reduce fracture strength.

*Allan Schrøder Pedersen*

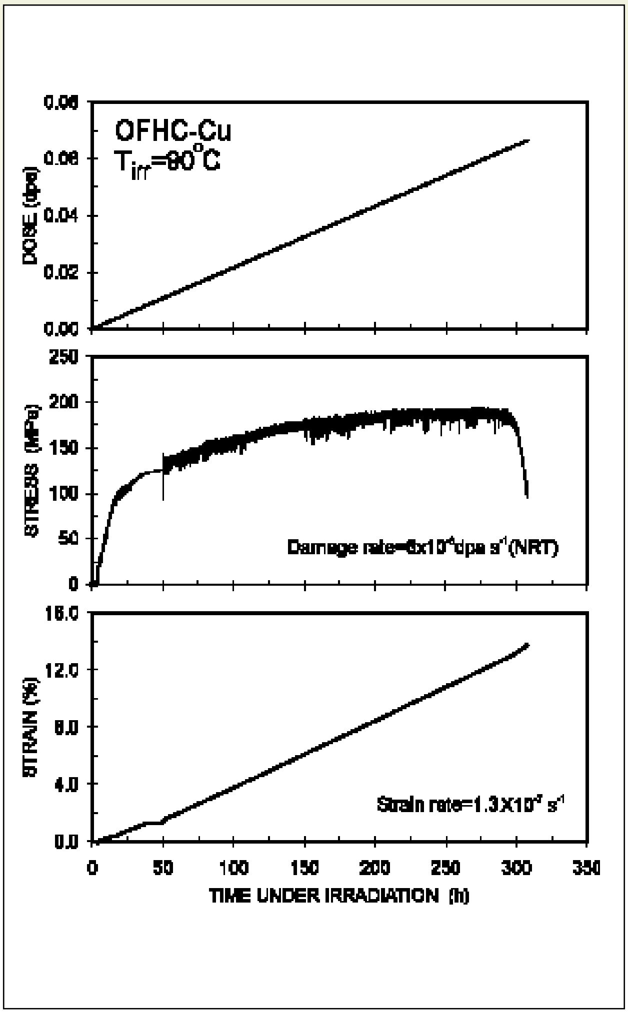
*Test assembly containing specimen and instrumentation for measuring stress, strain, temperature and neutron fluence.*

## In-Reactor Tensile Test of Pure Copper

It has been a common practice for more than 40 years to assess the adverse effects of neutron irradiation on mechanical performance of metals and alloys on the basis of the results of post-irradiation deformation experiments. In these experiments, the specimens are first irradiated to a certain dose level in unstressed condition (i.e. in the absence of continuous dislocation generation) and then mechanically tested outside of the reactor (i.e. in the absence of the continuous production of defects). These experiments have shown that the neutron irradiation causes a drastic decrease in the ductility of the irradiated materials, and the results have raised serious problems in evaluating the lifetime of materials used in structural components of a fission or fusion reactor.

Materials employed in structural components of a reactor, on the other hand, will experience simultaneously external stresses and continuous defect generation by neutrons. Recent theoretical considerations suggest that under these conditions the deformation behaviour may be substantially different from that observed in post-irradiation experiments. In order to resolve this issue, we have recently determined the dynamic stress-strain curves for pure copper subjected simultaneously to plastic deformation and neutron irradiation in the BR-2 reactor at Mol in Belgium. Up to date, no such experiment has been reported in the literature. The results clearly demonstrate that the deformation behaviour in the in-reactor experiment is fundamentally different from that observed in the post-irradiation experiments. Furthermore, the in-reactor experiments do not show any sign of a yield drop, plastic instability or a drastic decrease in the uniform elongation of the material as suggested by post-irradiation experiments.

*Bachu Singh*




*Dynamic stress response of the specimen in the test module as a function of irradiation time showing the effect of radiation damage and external strain acting simultaneously.*

## New JEOL 3000F Transmission Electron Microscope

A new JEOL 3000F transmission electron microscope (TEM) has been installed at the Materials Research Department. The microscope was funded by the Research Council Programme “Expensive apparatus”, by the Technical-Scientific Research Council, and by Risø National Laboratory. It is a national centre facility with access for all Danish TEM users. The centre participants are the University of Copenhagen, the University of Aarhus and the Technical University of Denmark.

The microscope is equipped with a field emission gun (FEG). Among the accessories are:

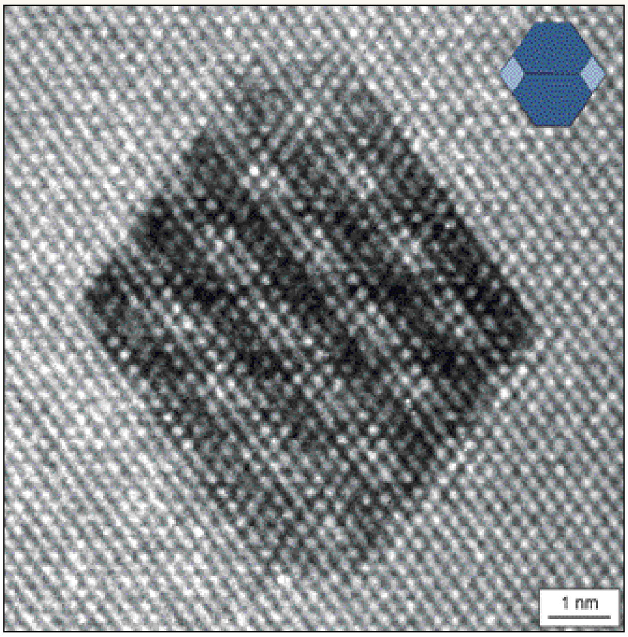
- energy image filter (EFTEM/GIF)
- parallel electron energy loss spectrometer (PEELS)
- energy-dispersive X-ray spectrometer,
- scanning transmission unit (STEM)
- high-angle annular dark field detector (HAADF)
- CCD cameras
- hollow-cone illumination
- piezo-stage based auto drift compensation system
- single-tilt high temperature stage

The microscope has a point resolution of 0.19 nm, an information limit of 0.10 nm and the spatial resolution for chemical analysis is under optimal conditions 0.5 nm.

The microscope was inaugurated on 19th September in the presence of approximately 80 invited guests. The lectures at the inauguration were given by Dr. R. Ravelle-Chapuis, JEOL France, by Dr. R. Wallenberg, University of Lund, by Dr. J. Hutchison, University of Oxford, and by Dr. P. Midgley, University of Cambridge.

An atomic resolution image of a lead inclusion embedded in an aluminium matrix is shown as an example of the applications of the microscope.

*Jørgen B. Bilde-Sørensen*




*An atomic resolution image of a lead inclusion embedded in an aluminium matrix. The dark lines are moiré fringes that arise due to the overlap in the image between the atomic planes of lead and aluminium. The insert shows the 3D shape of the lead inclusion in form of an octahedron with truncated corners.*

*Modern wind turbine blades, made of fibre composite materials, are strong and can sustain large deflections without fracturing (picture shown by permission of Vestas A/S).*

# Composites and Materials Mechanics

The programmes Materials Mechanics and Composite Materials were from 1st January 2003 merged into a joint programme. The following gives a short description of the new programme, its objectives and profile.

The programme is active in scientific and applied materials research and development. The effort is directed towards research, innovation, and education; the engineering focus is on energy and industrial applications of composite materials.

Main objectives are to develop, characterize and improve composite materials based on metals, polymers, and ceramics for energy purposes and other industrial applications. The goal is to extend the knowledge of light and strong materials seen from both a functional point of view and an application point of view. The work is focused on optimising properties, developing new materials, using conventional methods and seeking new methods for manufacturing, destructive and non-destructive characterization, and numerical modelling. The work is carried out in close contact and collaboration with national and international industrial partners, the education sector, and the research sector.

## Highlights of the year include

*Surfaces and interfaces*
A new post. doc. project was started with the aim to analyse and estimate the effect of fibre surface sizing on glass fibre reinforced polymers. The project is carried out in close collaboration with the Danish Polymer Centre at Risø.

*Filament winding of Fibre Reinforced Plastics (FRP)*
A special filament winding technique was used to manufacture flat unidirectional laminates for purpose of characterization of different fibre/matrix combinations. The technique was used for laminates with thermosetting matrices as well as for thermoplastic matrices. In the case of a thermosetting matrix, the fibres are wound dry onto a frame, and the matrix is subsequently applied by a resin infusion technique. In the case of a thermoplastic matrix, the structural fibres and thermoplastic fibres are co-wound onto the frame, and the laminate is formed in a subsequent press consolidations process.

*Autoclave processing of FRP*
Autoclave consolidation of thermoplastic composites is a versatile process to manufacture high quality laminates from different types of semi-raw products. This technique was used to manufacture various laminates of thermoplastic composites with natural fibres.

*Press consolidation of FRP (thermoplastic polymers)*
Processing technologies of products for Danish industrial parties were developed successfully.

*Organic fibre and matrix materials*
The natural fibre composites were placed in the context of sustainability at the Risø symposium and the first industrial enterprise was initiated with a Danish company.

*Numerical modelling of damage properties*
Cracking along the skin/core interface in sandwich specimens under general edge loads was analysed. The problem was solved analytically except for a single scalar load-independent parameter that was determined from finite element analysis. Results were developed for a wide range of elastic mismatch and core and skin thickness ratios.

*Mechanical properties, reliability, endurance and life-time*
In the EU 5th frame programme project COLT on the cyclic behaviour of high strength tool steel, a lifetime prediction model was suggested and verified.

*Tribology and wear*
Biaxial mechanical test equipment for determining wear properties of biotech materials was installed and tests were run successfully .

*Ballistic properties and protection material*
The EUCLID 3.19 project on operational aspects of add-on-armour was finished successfully with one year of field conditioning in Denmark followed by a shooting test.

*Structural Health Monitoring*
Piezoelectric acoustic emission sensors and fibre optic micro bend sensors have been successfully used to monitor structural condition during full scale testing of wind turbine blades. A remotely operated and structurally integrated system is to be developed for use on blades during standard operation. Support has been provided to European collaboration projects developing structural health monitoring solutions for Aerospace and Naval platforms.

*Ultrasound, X-ray, Acoustic Emission, Thermography*
An air-coupled ultrasonic system was installed. This enables scanning without water coupling and is especially useful for sandwich materials. The scanning area is up to 1.5 x 1 $m^2$.

*Fracture mechanics and resistance (R) curve behaviour*
A specimen for general mixed mode cracking was developed. The specimen is a double cantilever beam specimen loaded with uneven bending moments. A special test fixture was also developed.

*Design of sandwich constructions*
Sandwich T-joint with improved strength for naval ships was designed and tested.

These highlights were reached in both national and international basic research, programme research and industrial research projects of both scientific and technological nature. The target groups and funding sources are found within the authorities, the research community, the educational system, and industry.

The activities also involve commercial research and development projects of innovative character. In these cases a strict confidentiality is kept between the contractors as the results are of competitive nature.

*Povl Brøndsted*

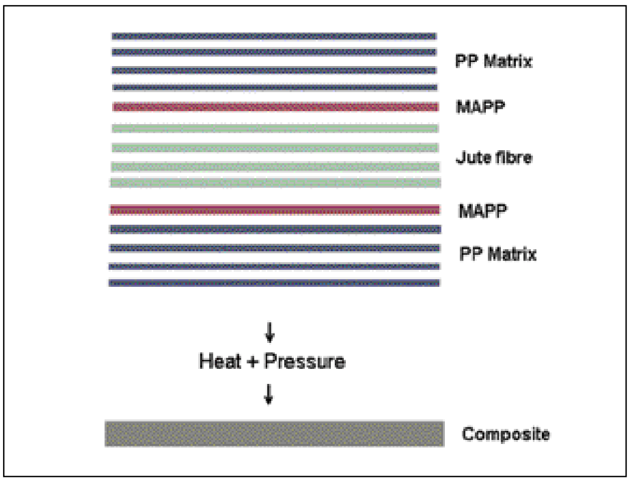
## Composites Research

Within the Composites and Materials Mechanics programme several on-going composite research projects on Ph.D., post doctoral, and industrial research level are relying on solid expertise in a wide range of composite manufacturing processes. A post doctoral research project was established in collaboration with the Danish polymer centre and an industrial partner, LM Glasfiber A/S, to gain better understanding of the influence of chemical modification of the glass fibre / matrix interface on macroscopic composite properties. For glass fibres, these interface properties are determined by the sizing, which is applied to the glass fibres during manufacture. Using commercial fibres with different resin systems, composite systems with weak and strong interfaces were identified and manufactured. This leads to a variation in macroscopic properties, of which the transverse strength (i.e., loading transverse to the reinforcement direction) gives a very good indication regarding interfacial strength.

Processing quality becomes especially important when composite differences on the microscale level are to be attributed to interface changes. Voids, which are easily introduced during manufacture, therefore have to be kept at a minimum. Of the available manufacturing techniques, dry filament winding (see left figure) followed by resin impregnation and autoclave cure or vacuum infusion, where resin is pulled into the sealed composite mould by pressure differences, are found to be suitable methods. The chosen manufacturing method obviously has to depend on the fibre and resin systems, and optimisation with respect to a certain system has to be performed in most cases.

New manufacturing techniques have to be developed for natural fibre composites. Natural fibre mats in combination with

*Stefanie Feih examines the precision of the computer controlled dry filament winding of a glass fibre composite plate.*




*Film stacking and in situ surface modification applying maleated polypropylene (MAPP) resulting in jute fibre reinforced polypropylene (PP) composite.*

thermoplastic polymer films potentially offer a rapid and simple means of manufacturing composites through film stacking, heating and press-consolidation. Work in this area within the programme lead to an invention and the international patent WO 02/064670 A1, which became publicly available on the 22nd of August 2002 (illustrated on previous page - right figure). The invention provides a number of advantages over the prior art, including one step in situ compatibilisation of fibre mats, improved processing flexibility for the manufacturer, easy handling/automation of compatibilisation treatment and lay-up process and a better performance of the composite material. The tensile strength of a jute/polypropylene (PP) composite with random fibre orientation was improved from 65 MPa to 86 MPa, similarly the tensile strength of a hemp/PP composite was improved from 40 MPa to 60 MPa and a flax/PP composite from 49 MPa to 69 MPa.

In relation to the press-consolidation technique, the compressibility of a fibre assembly is an important factor, which limits the fibre content of the manufactured composite. In an ongoing Ph.D. project it was found that glass fibres were more easily compressed than natural fibres and moreover, the random arrangement of fibres in a mat product severely reduced the compressibility. These results indicate that when the reinforcement potential of a given fibre material is evaluated, the spatial structural organization of the fibres must be considered in addition to the mechanical properties.

*Tom Løgstrup Andersen, Stefanie Feih and Bo Madsen*

## X-ray non-destructive characterization

X-rays give access non-destructively to the internal structures of a material (composition, presence of defects etc.). Our research activities are concerned with non-destructive characterization by x-ray imaging: prototype characterization, image processing, defect detection, damage characterization, and materials characterization in relation to the mechanical properties. The techniques commonly used are radiography, radioscopy and spectrometry.

### Radioscopic system

Radioscopic devices are now preferred to film radiography in a number of industrial application areas. A key interest of a real-time imaging system is to be able to optimise the conditions of acquisition and the projection while moving the imaged object in front of the detector. Another interest is the possibility of automatic defect detection, i.e. for on-line process control. A radioscopic system was set-up and developed at Risø to allow convenient routine examination of samples. The system involves an image intensifier and a manipulation unit with a 3-axis-translation and 1-axis-rotation table. The inspection system has been designed to cover a large range of applications.

### X-ray inspection of aluminium castings

The aim of the EU funded project QUME is to improve the performance of inspection of aluminium cast parts by data fusion of information coming from several non-destructive testing methods. Our role is to develop quantitative x-ray characterization methods by combining two approaches: radioscopic inspection using an image intensifier and spectrometric measurements.

Indeed, cast components have usually a complex shape with varying thickness. For safety parts, a 100 % x-ray inspection is often required. High thickness (typically > 40 mm) is difficult to inspect by radioscopy and the defect detection performance is very low in this range. Spectrometry is not an imaging tool, but a method potentially sensitive to small thickness or density variations.

The spectrometry system involves a NaI photon-counting detector and a multi-channel analyser. Each measurement corresponds to a spectrum of number of photons (intensity) transmitted through the sample at this point, as a function of their energy.

Our spectrometric system showed a higher sensitivity in thickness than radioscopy and spectrometry is therefore used as a complementary method to radioscopy for areas in cast pasts of high thickness.

*Emmanuelle Cendre*

*Emmanuelle Cendre is mounting a specimen in the real-time radioscopic inspection system.*

*The spinning polishing plate of the new Logitech PM52 lapping and polishing machine, which allows polishing samples down to a pre-specified depth with an accuracy of one micrometer. This precision allows us to polish down to an interesting area in a sample, which has been characterised by other experimental bulk techniques (such as 3DXRD microscopy). This means that we can compare, and even combine experimental techniques, allowing us to perform experiments that we would otherwise not be able to - such as nucleation studies in polycrystalline metals.*

# Metal Structures in Four Dimensions

The overall objective of the work in the programme is to explore the heterogeneous structure of metallic materials and understand how this responds over time to changes in stimuli such as stress and temperature. We seek to derive fundamental theories and formulate models for the structural development within materials. This involves experimental characterization and modelling on several length scales.

The vast majority of the financing comes from the Danish National Research Foundation establishing us as a Centre for Fundamental Research. The Contract period runs from 1st August 2001 to 30th June 2006.

### Scientific highlights of the year include

- Demonstration of an arithmetic reconstruction method to analyse 3-Dimensional X-Ray Diffraction (3DXRD) data, which improves the spatial resolution from 25 µm to 5 µm.

- Development and documentation of a routine to extract the exact position (x, y, z) of marker particles from microtomography data and to follow their motion during, for example, deformation. This has potential applications in many fields of materials science.

- Nanoscale structures are achieved via deformations to very high strains. However, it is shown that even after a strain as large as 10, correlation still exist between neighbouring structure elements, i.e. the original grains are remembered.

- Strong evidence is provided that nuclei with new orientations relative to the deformed matrix may develop during recrystallisation. This is very controversial and against common modelling practice.

Other highlights have been a very active guest programme, including external users of the 3DXRD equipment. The latter has lead to a joint publication in Science on phase transformations. Also, the Summer School in China on "Geometry of Microstructures" organised together with Tsinghua and Yanshan Universities was, indeed, a highlight.

*Dorte Juul Jensen*

*Larry Margulies receiving the European Synchrotron Radiation Facility's Young Scientist Prize.*

## Summer School and Symposium

A Summer School on Geometry of Microstructures and the First Joint Danish-Chinese Materials Science Symposium on Characterisation of Microstructures were held in Qinhuangdao in China during the period 16th – 20th of August 2002 organized by the Centre of Fundamental Research: *Metal Structures in 4D* in collaboration with Tsinghua and Yanshan Universities. During the School, experienced lecturers from China, Poland and the United Kingdom provided the necessary background on theory, analysis methods as well as techniques. The Symposium subsequently documented the use of these tools at an international research level. This combination worked perfectly and was fruitful to all 65 participants: 10 Danish students from DTU, KU, AU and AAU, 7 Danish students and 8 scientists from the Centre, 22 Chinese students and 11 scientists from 8 universities.

*Wolfgang Pantleon*

*Participants from the Summer School on Geometry of Microstructures and the First Joint Danish-Chinese Materials Science Symposium on Characterisation of Microstructures sitting on China's greatest macro-structural feature, where it meets the sea.*

## Measurements of Plastic Strain in 3D

A project was initiated in 2001 with the goal of using X-ray absorption micro-tomography to detect marker particle displacements inside a metallic material undergoing plastic deformation. The displacements of marker particles as a function of externally imposed strain are then used to deduce the internal strain in local regions.

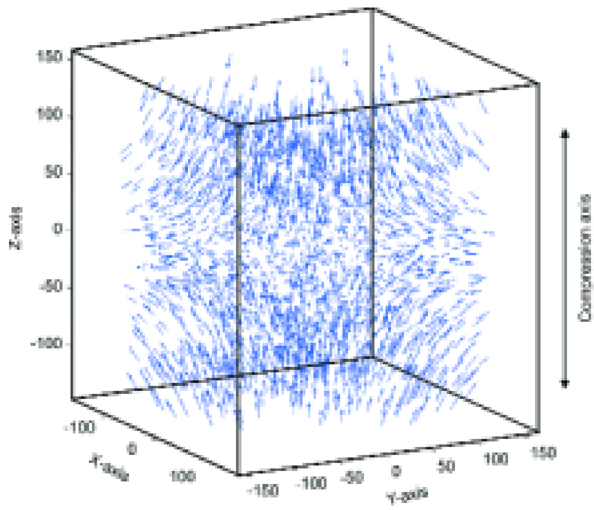
After a series of image analysis steps to identify the centre of mass of individual particles in the tomographic reconstructions, the displacements of individual particles could be tracked as a function of external strain.

The particle displacements are then used to identify local displacement gradient components, from which the local 3D plastic strain tensor can be determined. This allows us, for the first time, to map the strain components as a function of location inside a deforming metallic solid.

A universal method has been demonstrated for bulk materials that contain particles or voids observable by X-ray tomography. The method has been demonstrated using an Al alloy containing W particles. The strain resolution is $10^{-2}$ on the measurements of displacement gradient components while the spatial resolution depends on particle spacing, in the present case 30 µm.

In upcoming stages of the project, we will use this technique to evaluate the effect of local grain orientation on local plastic strain, allowing us to test 6 decade-old theories of metal deformation that remain incompletely tested. We also envision that this technique can be used to study deformation of foams, strain fields around cracks, deformation of biological materials, etc. Since the technique does not depend on diffraction, the evaluation can be applied to any material, crystalline or amorphous, to which marker particles providing sufficient X-ray absorption contrast have been introduced. We believe that the technique we have demonstrated provides an avenue for quantitative analysis of micro-tomography images with a great number of possible applications.

*Søren Fæster Nielsen*



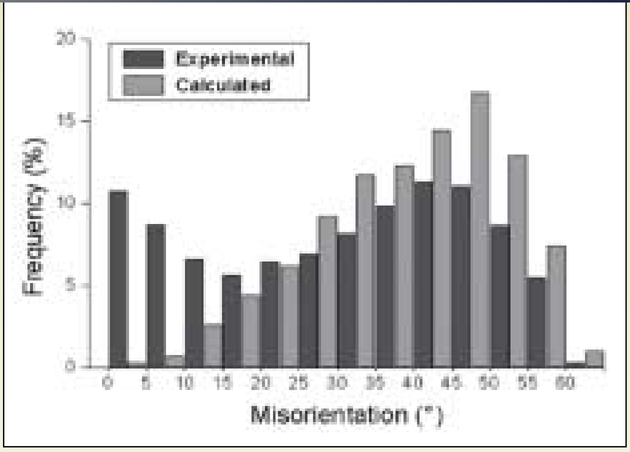

*The vector displacement tracks of 2544 particles within a 0.4 mm cube inside the sample. The numbers on the axes are in units of pixels of 1.5µm.*

### Finer 3DXRD Resolution

A novel method based on an Algebraic Reconstruction Technique for the generation of three dimensional grain maps has recently been developed. Both experimental validation and computer simulation results have revealed that this technique is so far the most efficient method for reconstruction of the grain map by 3Dimensional X-Ray Diffraction (3DXRD) microscope. It reduces the spatial resolution from previous 25 µm down to 5 µm.

*Xiaowei Fu*

## Nanoscale Microstucture Investigations




*The distributions of misorientation angles between selected orientations; neighbouring orientations in dark grey (experimental data) and non-neighbouring in light grey (calculated data). The difference between the two indicates the presence of orientation correlations.*

Metallic materials composed of nanoscale grains are interesting due to their potentially advanced properties. One possible route to produce them is high strain deformation. This processing route is favourable as it provides fully dense materials. Investigation of the deformation processes that occur during the creation of these microstructures is therefore of fundamental importance.

Typically strains of the order of 10 are required to induce sufficient grain fragmentation to the sub micrometer scale. This is equivalent to rolling a 1m thick slab of metal to kitchen foil (50 µm thick). Recent analysis of neighbouring grain orientations, determined by electron backscattering, has been performed on an aluminium alloy after such deformation. This material contains on average 0.3 x 0.8 µm sized grains with a near random texture, which prior to deformation had a coarse grain size and strong texture.

Analysing the orientations of neighbouring grain fragments has shown that after such a massive strain, correlations still exist between them, suggesting that the microstructure still retains a memory of its deformation history despite, being randomised by the deformation.

*Jacob R. Bowen*

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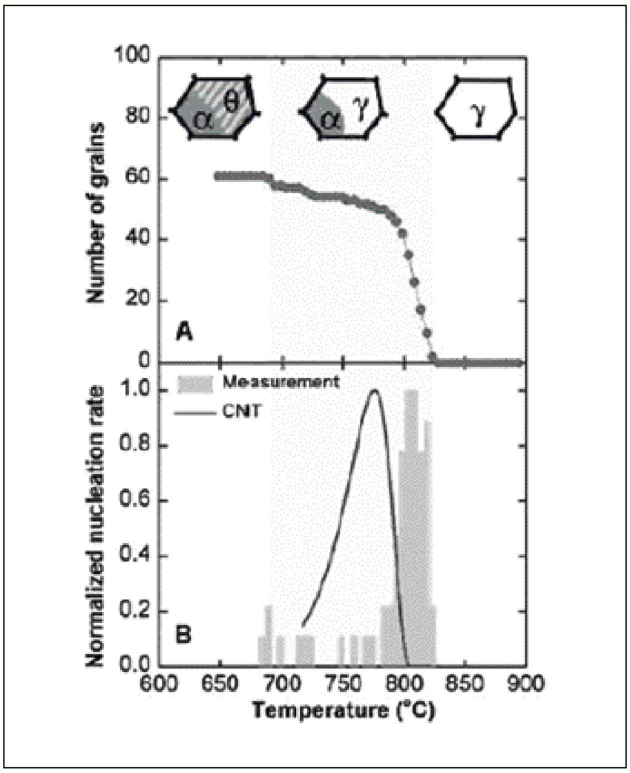
## Grain Nucleation and Growth During Phase Transformations in Steel

Steel is one of the most important materials used in thermo-mechanical processing and is used extensively in a broad range of industrial applications. Despite this fact, the fundamental mechanisms for nucleation and growth in these materials are still poorly understood.

In collaboration with S.E. Offerman and co-workers from Delft University of Technology and the European Synchrotron Radiation Facility (ESRF), Grenoble, France, *in-situ* studies of the kinetics of phase transformations in steel has been performed. The experiment was performed using the 3-Dimensional X-ray Diffraction (3DXRD) microscope located at the Materials Science beam line at ESRF.

The analysis of the synchrotron data has provided two remarkable sets of experimental results on the austenite-to-ferrite phase transformation in steel. One is the *in-situ* observation of the nucleation rate during cooling. The second set of observations follows the growth of individual ferrite grains as a function of temperature and time. Based on these observations valuable information regarding the activation energy for nucleation and the interaction between grains during growth has been obtained.

*Erik M. Lauridsen*




*Nucleation as a function of temperature during continuous cooling of the steel from 900°C to 600°C in 1 hour. (A) The total number of valid ferrite reflections. (B) The normalized experimental nucleation rate (grey bars) compared to the classical nucleation theory (line).*

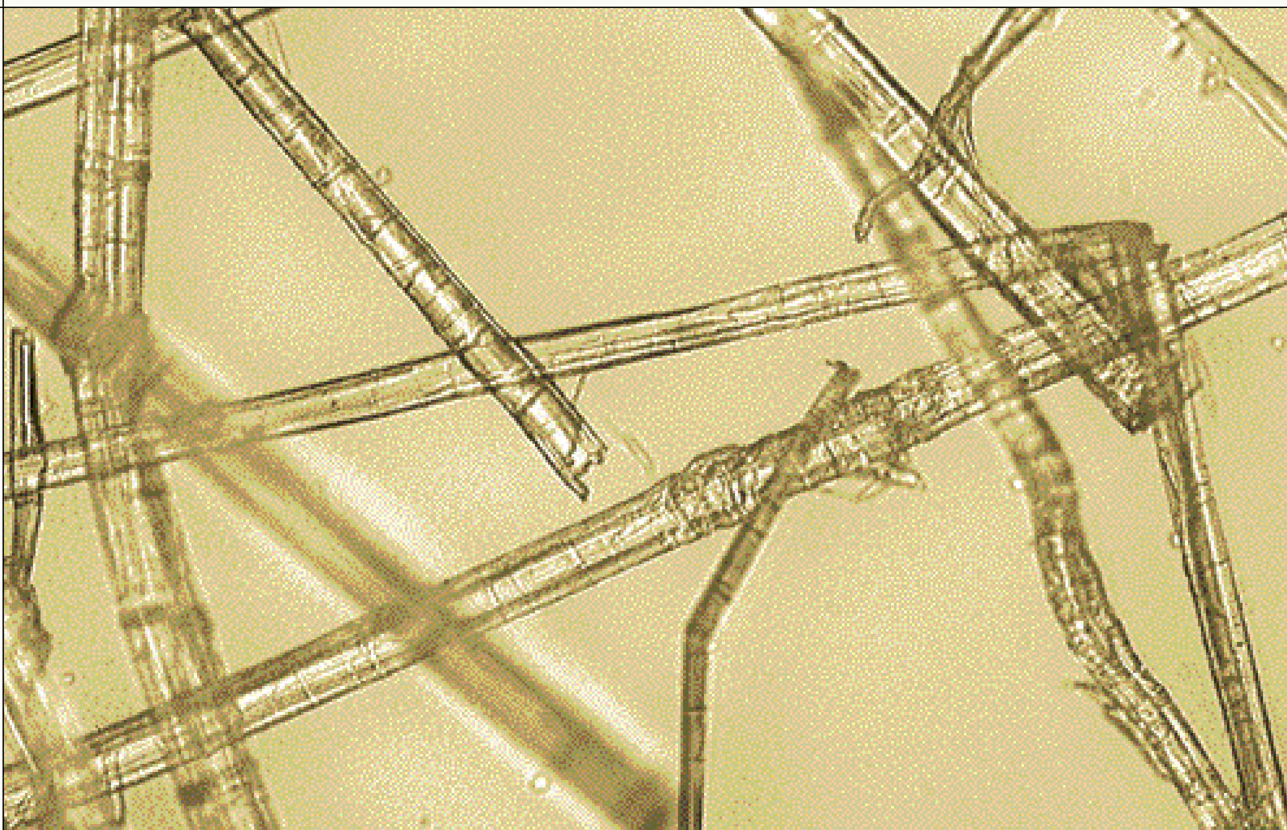
# 23rd Risø International Symposium on Materials Science

The Symposium "Sustainable Natural and Polymeric Composites – Science and Technology" was held at Risø on 2nd – 5th of September 2002. The focus of the Symposium was on the potential for exploiting natural fibres for strong composite materials, and at the same time looking at the possibilities of using polymers in a sustainable way as a matrix in composites. The field of topics was covered by 7 invited key-note lectures and 28 contributed papers, addressing materials systems, properties and processing.

Several developments and new possibilities were presented: Optimised hot pressing of long fibre thermoplastics made of jute and polypropylene; performance of single and bundled natural cellulose fibres; deformation micromechanics of natural fibres; nanotechnology in relation to polyurethane with cellulose fibres. The combination of sustainable concepts and materials and energy was presented in a vision of a "biomass refinery" producing bio fuels, natural polymers and natural cellulose fibres.

The participants, numbering about 60, took part in lively discussions. Several new ideas emerged, e.g. the potential for very high strength cellulose based on nanoscale crystallites. New personal network contacts were established and existing ones renewed during the Symposium. The four days of its duration allowed for new initiatives to be discussed and developed.

*Hans Lilholt*

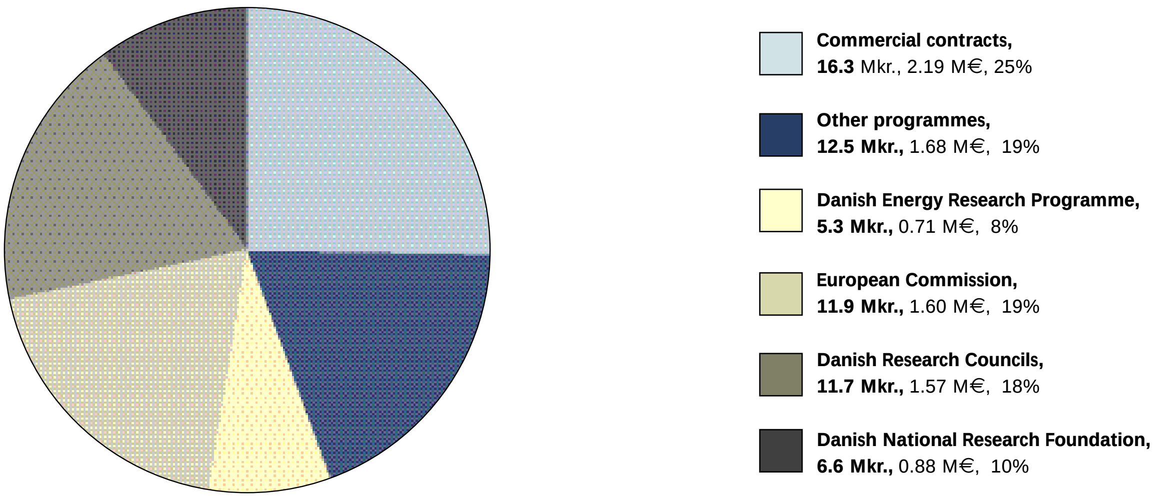


*Hemp fibres are important natural fibres for strong composite materials. They have a high content of cellulose and form long fibres. Parallel and long fibres give strong composites, but only if fibres are strong and without defects. The picture shows individual hemp fibres observed in the optical microscope. The fibres are rather long and straight, but contain some defects (markings across the fibres). The diameter of the individual fibres is about 20 micrometers. Work is in progress to treat hemp fibres more gently in order to reduce the number of defects.*

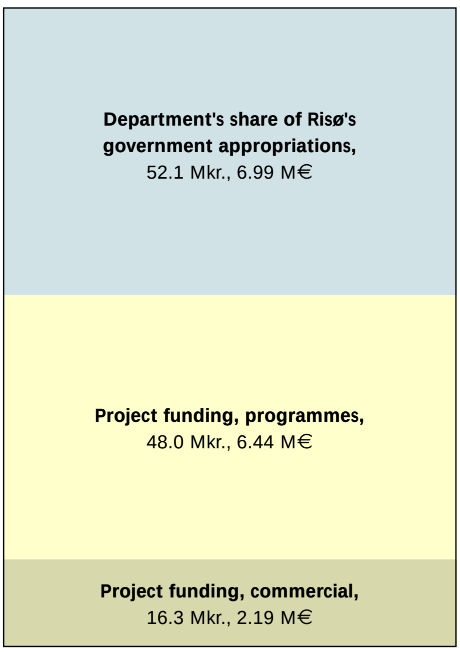
# Finances

The activities of the Department are supported by a combination of government funding, focused project funds from national and international research programmes, and fully commercial contracts.

## Project Income

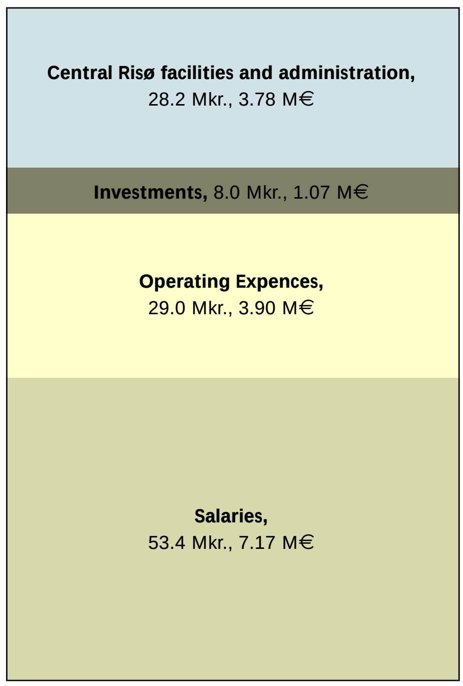



## Income




**Total:** 116.4 Mkr., 15.6 M€

## Expenditures




**Total:** 118.6 Mkr., 15.9 M€

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Andersen, Niels Hessel
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Jørgensen, Thomas
Nielsen, Heidi Kolmorgen
Nyrup, Susan Blak
Søgaard, Martin
Thorning, Casper

**Emeritus**
Hansen, Niels
Nielsen, Mourits

Persons leaving (#) or joining (*) the Department during 2002. (§) Employed at University of Copenhagen.

*A new open space office has been created to house the increasing number of young scientists.*

# Educational work

The Department is strongly involved in the education of students at different levels. The involvement ranges from postgraduate and undergraduate courses and projects, in collaboration with universities and industry, to lectures for high school classes. Several international workshops and summer schools aimed at Ph.D. students have been arranged. In addition many staff members of the Department act as external university lecturers and examiners. The education of Ph.D. students and that of Masters Degree students are some of the most important educational activities. During 2002 four Ph.D. students completed their Ph.D. projects, while 23 projects were still ongoing, in addition to 12 Masters Degree projects.

## Ph.D. projects completed in 2002

**Karin Vels Hansen**
”Interface in composite electrodes for SOFC and high temperature electrolysers”. Technical University of Denmark. Supervisors: Ib Chorkendorff (DTU), Mogens Mogensen, Jørgen B. Bilde-Sørensen.

**Søren Koch**
“Contact resistance of ceramic interfaces between materials used for solid oxide fuel cell applications”. Technical University of Denmark. Supervisors: Torben Jacobsen (DTU), Peter Vang Hendriksen, Mogens Bjerg Mogensen, Carsten Bagger.

**Eva Ravn Nielsen (Industrial Ph.D.)**
Ferroperm Piezoceramics A/S, “Low temperature sintering of PZT” Technical University of Denmark. Supervisors: Kenny Ståhl (DTU), Finn Willy Poulsen (Risø), Erling Ringgaard (Ferroperm Piezoceramics A/S).

**Séverine Ramousse (Industrial Ph.D.)**
Obtec A/S, “Development of high temperature stable friction materials” Technical University of Denmark. Supervisors: Irina Petrushina (DTU), Ole Toft Sørensen (Risø), Jesper Valentin (Obtec A/S).

## Ongoing Ph.D. projects

**Asger Abrahamsen**
“Magnetic properties of superconductors”. Technical University of Denmark. Supervisors: Jørn Bindslev Hansen (DTU), Claus Schelde Jacobsen (DTU), Niels Hessel Andersen.

**Anders Andreasen**
“Preparation and characterization of new metal hydrides for hydrogen storage”. Technical University of Denmark. Supervisors: Ib Chorkendorff, (DTU), Søren Dahl (Haldor Topsøe A/S), Flemming Besenbacher (AU), Allan Schrøder Pedersen.

**Jens W. Andreasen**
“Nano-structured catalysts”. Technical University of Denmark. Supervisors: Kenny Ståhl (DTU), Frank Berg Rasmussen (Haldor Topsøe A/S), Robert Feidenhans´l.

**Niels Bech Christensen**
“Quantum phase transitions”. University of Copenhagen. Supervisors: Jens Als-Nielsen (KU), Desmond F. McMorrow.

**Jesper Christiansen**
“Dislocation interactions with surfaces and grain boundaries”. Technical University of Denmark. Supervisors: Karsten Wedel Jacobsen (DTU), Jakob Schiøtz, Torben Leffers.

**Anders Reves Dinesen**
“Magnetic properties of amorphous and nanocrystalline alloys”. Technical University of Denmark. Supervisors: Steen Mørup (DTU), Søren Linderoth, Nini H. Pryds.

**Carsten Gundlach**
”Recovery in aluminium”. University of Copenhagen. Supervisors: Erik Johnson (KU), Henning Friis Poulsen, Wolfgang Pantleon.

**Anette Nørgaard Hansson**
“Protection against metallic oxidation”. Technical University of Denmark. Supervisors: Marcel Somers (DTU), Søren Linderoth, Mogens Mogensen, Peter Friehling.

**Jens V. T. Høgh**
“Kinetics of metal/hydrogen electrode on solid electrolytes”. Technical University of Denmark. Supervisors: Torben Jacobsen (DTU), Ib Chorkendorff (DTU), Mogens Mogensen.

**Christian Højerslev**
“The influence of microstructure on the fatigue properties of high strength materials for cold forging tools”. Technical University of Denmark. Supervisors: Marcel Somers (DTU), Povl Brøndsted, Jesper Vejlø Carstensen.

**Stine Nyborg Klausen**
”Magnetic dynamics of nanoparticles”. Technical University of Denmark. Supervisors: Steen Mørup (DTU), Kim Lefmann, Kurt N. Clausen.

**Trine Klemensø**
”Relationships between structures and performance of SOFC anodes”. Technical University of Denmark. Supervisors: Torben Jacobsen (DTU), Jørgen Gutzon Larsen (Haldor Topsøe A/S), Mogens Mogensen.

**Axel W. Larsen**
”Nucleation in metals”. Technical University of Denmark. Supervisors: Jens Als-Nielsen (KU), Henning Friis Poulsen, Dorte Juul Jensen.

**Zhengjie Li**
“Anisotropy and dislocation structures at large strains”. Technical University of Denmark. Supervisors: Niels Bay (DTU), Grethe Winther, Niels Hansen.

**Bo Madsen**
”Properties of plant fibre composites – an experimental model study”. Supervisors: Lars Damkilde (DTU), Preben Hoffmeyer (DTU), Anne Belinda Thomsen (PRD, Risø), Hans Lilholt.

**Lars Mikkelsen**
”Oxidation of iron-chromium alloys”. University of Southern Denmark. Supervisors: Eivind Skou (SDU), Søren Linderoth, Mogens Mogensen, Peter Halvor Larsen.

**Trine Bjerre Pedersen**
”Modelling of residual stresses in sprayformed structures”. Technical University of Denmark. Supervisors: Jesper Hattel (DTU), Nini H. Pryds.

**Mette Poulsen**
“Nanostructuring with bonding”. Technical University of Denmark. Supervisors: Flemming Jensen (DTU), Robert Feidenhans´l.

**Henrik Nikolaj Blicher Schmidt**
”Modelling of mechanical and metallurgical properties of friction stir welded joints”. Technical University of Denmark. Supervisors: Jesper Hattel (DTU), John A. Wert.

**Martin Søgaard**
”Properties of perovskites with varying A/B-ratio”. Technical University of Denmark. Supervisors: Torben Jacobsen (DTU) , Peter Vang Hendriksen, Mogens Mogensen, Finn Willy Poulsen.

**Anders Thygesen**
“Hemp fibres for light and strong composites – optimisation and characterisation”. Royal Veterinary and Agricultural University. Supervisors: Per Ole Olesen (KVL), Claus Felby (KVL), Anne Belinda Thomsen (PRD, Risø), Hans Lilholt.

**Katrine Nørgaard Toft**
”Magnetic properties of superconductors”. University of Copenhagen. Supervisors: J. Jensen (KU), Niels Hessel Andersen.

**Christian Bech Wejdemann**
“Nanomechanics of metal fatigue”. Technical University of Denmark. Supervisors: Karsten W. Jacobsen (DTU), Ole Bøcker Pedersen, J. B. Bilde-Sørensen.

## Masters projects

**Peter Andersen**
“Antiferromagnetism in YBCO nanoparticles”. University of Copenhagen. Supervisors: Jens Als-Nielsen (KU), Luise Theil Kuhn.

**Anne Egebjerg**
“X-ray scattering investigations of bonded interfaces”. University of Copenhagen. Supervisors: Sine Larsen (KU), Flemming Jensen (DTU), Robert Feidenhans’l.

**Karen-Anne Herdahl**
”Exact numerical diagonalisation of magnetic Bloch oscillations in one dimension”. Roskilde University. Supervisors: Jeppe Dyre (RUC), Kim Lefmann.

**Jari í Hjøllum**
“Maghemite and hematite nanoparticles, synthesis and magnetic properties”. University of Copenhagen. Supervisors: Morten Bo Madsen (KU), Luise Theil Kuhn.

**Birgitte Abery Jacobsen**
“Critical currents in BiSCCO/Ag tapes for superconducting po-

wer cables". University of Copenhagen. Supervisors: P.E. Lindelof (KU), Jørn Bindslev Hansen (DTU), Niels Hessel Andersen.

**Anette Jensen**
"A neutron scattering study of the magnetic structures in superconducting $ErNi_2B_2C$". University of Copenhagen. Supervisors: Per Hedegård (KU), Niels Hessel Andersen.

**Søren Højgaard Jensen**
"High temperature solid oxide electrolyser". University of Copenhagen. Supervisors: Jan W. Thomsen (KU), Mogens Mogensen.

**Thomas Jørgensen**
"Investigation of the superconducting properties of $MgB_2$/Fe tapes". University of Copenhagen. Supervisors: Per Hedegård (KU), Jean-Claude Grivel.

**Heidi Kolmorgen Nielsen**
"Exact numerical diagonalisation of magnetic Bloch oscillations in one dimension". Roskilde University. Supervisors: Jeppe Dyre (RUC), Kim Lefmann.

**Susan Blak Nyrup**
"X-ray scattering from hair. A new method for detection of breast cancer?". University of Copenhagen. Supervisors: Jens Als-Nielsen (KU), Robert Feidenhans'l.

**Martin Søgaard**
"Investigation of transport properties and oxygen stoichiometry in $(La_{0,6}Sr_{0,4})_{0,99}Co_{1-y}Fe_yO_{3-y}$". University of Southern Denmark. Supervisors: Eivind Skou (SDU), Peter Vang Hendriksen, Mogens Mogensen, Finn Willy Poulsen.

**Casper Thorning**
"Orientation subdivision of grains of known orientation in polycrystals strained in tension". Technical University of Denmark. Supervisors: Marcel A.J. Somers (DTU), John Wert.

## Lecturing courses and other teaching activities

**Current topics in Physical Metallurgy**
Risø, autumn 2002. Lecturer: Roger Doherty.

**Eksperimentel Faststofmekanik**
DTU, course no. 41811, 2002. Lecturer: Bent F. Sørensen.

**ICFA Instrumentation School**
Istanbul, Turkey, Jun 17-28, 2002. Lecturer: Kurt Nørgaard Clausen.

**Korrosion af keramik og glas**
DTU, course no. 42135, 2002. Lecturer: Karsten A. Nielsen.

**Magnetism**
Course for Ph.D. and master students, NBI, spring 2002. Member of organising committee and lecturer: Kim Lefmann, Exercises: N.B. Christensen, Kim Lefmann.

**Nano Magnetism**
Study group for Ph.D. students and others, Risø, DTU and NBI, 2002 Lecturers: Stine N. Klausen, K. Lefmann, Per-Anker Lindgård, Luise Theil Kuhn.

**Nanoteknologi 1**
Course at University of Copenhagen, autumn 2002. Co-supervisor: Christian Bech Wejdemann.

**Nano Science Seminars**
for Ph.D. students and others, NBI, 2002. Member of organising committee: Luise Theil Kuhn.

**Physics and Chemistry of Nanostructures**
Course for Ph.D. and master students, DTU, autumn 2002. Lecturer: Luise Theil Kuhn.

**QUP-Seminars**
DTU, 2002. Lecturer: Per-Anker Lindgård.

**SOFC brændselsceller**
Thisted og Himmelev Gymnasium, 2002. Lecturer: Jesper Knudsen.

**SOFC brændselsceller**
Rysensteen Gymnasium, 2002, Lecturer: Rasmus Barfod.

**Summer School on Functional Ceramics**
KTH, Sweden, 2002. Lecturer: Dorthe Lybye.

**Tillämpad Kärnfysik**
Lunds Universitet, Sweden, autumn 2002. Lecturer: Kim Lefmann.

**X-ray Physics**
Course for Ph.D. and master students, NBI, 2002. External Lecturer: Desmond F. McMorrow.

# Prizes, awards, honours

**Morten Eldrup**
Appointed Adjunct Professor, Department of Polymer Science and Engineering, Lund Institute of Technology, Lund University, Sweden

**Karin Vels Hansen**
Received the A.R. Angelos award

**Lawrence Margulies**
Received the ESRF Young Scientist Prize

# Organization of international meetings

**Meeting of The Scandinavian Neutron Scattering Societies at the European Conference**
Bonn, Germany, May 15, 2002. Members of Organizing Committee: Kurt Nørgaard Clausen, Bente Lebech

**Nano Physics of Life Systems**
Carlsberg, Copenhagen, Denmark, Jun 21-22, 2002.
International conference, 80 participants.
Organizers: Per-Anker Lindgård, H. Bohr

**5th European SOFC Forum**
Lucerne, Switzerland, Jul 1-5, 2002, 400 participants.
International Advisory Committee: F.W. Poulsen

**Dynamics of Biological Molecules and Networks: From Single Molecules to Networks,**
Krogerup, Denmark, Aug 10-17, 2002.
Member of Organizing Committee: Henrik Flyvbjerg

**First Joint Chinese-Danish Symposium:**
Qinhuangdao, China,
**Characterisation of Microstructure**
Aug 19-20, 2002, and
**Summer School on Geometry of Microstructures**
Aug 16-18, 2002, 67 participants.
Members of Organizing Committee:
Jacob R. Bowen, Andy Codfrey, Wolfgang Pantleon

**International Conference on New Challenges in Mesomechanics**
Aalborg, Denmark, Aug 26-30, 2002.
International Advisory Committee: B.F. Sørensen

**23rd Risø International Symposium on Materials Science: Sustainable Natural and Polymeric Composites Science and Technology**
Risø National Laboratory, Denmark, Sep 2-5, 2002.
Organizers: H. Lilholt, Bo Madsen, Helmuth Toftegaard, E. Centre, Modris Megnis, Lars P. Mikkelsen, Bent F. Sørensen

**5th Topical Meeting on Biophysics-Biological Physics**
Copenhagen, Denmark, Nov 26, 2002
Organizer: Henrik Flyvbjerg

**Mini-workshop on Protein Aggregation**
Copenhagen, Denmark, Dec 6, 2002.
Member of Organizing committee: Henrik Flyvbjerg

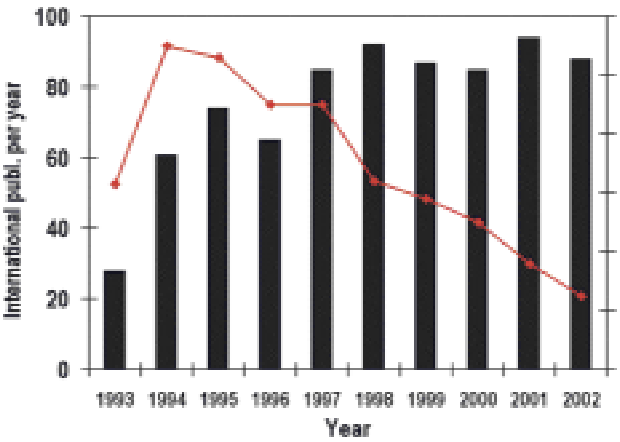
**Joint Nordic Conference in Powder Technology**
Tampere, Finland, Feb 5-6, 2003.
Member of Organizing Committee: A. Schrøder Pedersen

# The Engineering Science Centre for Structural Characterization and Modelling of Materials

Engineering science centres are the largest single commitments of the Danish Technical Research Council (STVF). Such a centre is first granted for five years with the possibility of an extension for another five years if the council finds it worthwhile. At the Materials Research Department we have been so fortunate as to host an engineering science centre, the Engineering Science Centre for Structural Characterization and Modelling of Materials, for ten years – from primo 1993 to ultimo 2002. The mechanical behaviour of materials has been the focal point of the centre.

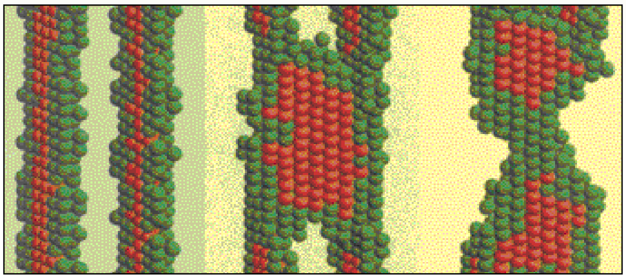
A grant for an engineering science centre requires that the host organization makes a contribution to the centre of at least the same magnitude as the contribution from STVF. In our case the total contribution from STVF has been ~61 million kr. (1 kr. = 0.13 Euro), and Risø has provided more than twice this amount. The contribution from Risø has comprised a direct contribution and a contribution via other contracts which have been part of the centre activities.

For STVF the basic philosophy behind the 5/10 years grants for engineering science centres is to boost research activities which should then be "anchored" in the host organization so that they can continue after the centre has expired. We think we have lived up to this philosophy as illustrated by the graph showing the number of international publications (including conference proceedings) from the engineering science centre for the ten years together with the contribution from STVF (corrected for inflation): during the ten years the number of publications has increased very significantly to reach an approximately constant level of ~90 publications per year while the contribution from STVF has been decreasing, particularly in the last five years. A natural extrapolation would be that the number of centre-relevant publications will remain constant even when the contribution from STVF decreases to zero.




One of the reasons for this successful anchoring is a grant from the Danish National Research Foundation for a Centre for Metal Structures in Four Dimensions for five years starting from August 2001 (with possible extension for another five years). The engineering science centre has played a very significant role in providing the basis for this new centre. Another new centre at the Materials Research Department, which to a large extent is based on the activities in the engineering science centre, is the centre for electron microscopy (together with Technical University of Denmark, University of Aarhus and University of Copenhagen).

The research themes of the engineering science centre have covered a wide spectrum of activities ranging from basic research like atomistic simulation of the cross-slip process and investigation of the deformation of polycrystals by modelling and experimental investigation by electron microscopy and diffraction by synchrotron X-rays to practical engineering studies of materials for wind-turbine blades and tools for forging.



As an example we show three frames from a molecular-dynamics study of the annihilation of a screw-dislocation dipole by cross slip. Green atoms are part of the cores of the Shockley partial dislocations, red atoms are part of stacking faults while atoms in perfect lattice positions are invisible. In the next frame the annihilation would be completed and all atoms would be invisible. This work was carried out in collaboration with the CAMP centre at Department of Physics at the Technical University of Denmark.

During the ten years we have been assisted by a National Centre Committee, and we want to thank those who have been members: R. Feidenhans'l, N. Hansen, K.W. Jacobsen, D. Juul Jensen, P. Madsen, N.-J. Rishøj Nielsen, J.K. Nørskov, J. Olsson, P. Pedersen and V. Tvergaard. We also want to thank the members of our International Advisory Board: H. Mughrabi, A. Thölén, V. Tvergaard and J.R. Weertman.

*Torben Leffers*

*Research and technology conquer the world - from the north shore of Risø.*

# Published work

## International Publications

*Andersen, L.G.; Poulsen, H.F.; Abrahamsen, A.B.;* Jacobsen, B.A.; Tschentscher, T., Microstructural dynamics of Bi-2223/Ag tapes annealed in 8% $O_2$. *Supercond. Sci. Technol.* (2002) v. 15 p. 190-201

Andrianov, A.V.; Iliushin, A.S.; Kosarev, D.I.; Zasimov, V.S.; *Lebech, B.*, Interplay of crystalline and magnetic structures in $Tb_xTm_{1-x}$ system. *J. Magn. Magn. Mater.* (2002) v. 251 p. 25-28

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Barlow, C.Y.; *Hansen, N.; Liu, Y.L.*, Fine scale structures from deformation of aluminium containing small alumina particles. *Acta Mater.* (2002) v. 50 p. 171-182

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Brooks, J.W.; *Sabin, T.J.*, A comparison of the macromodel and neural network approaches for the prediction of microstructural evolution during hot forging. *Proc. Inst. Mech. Eng. B* (2001) v. 215 p. 915-925

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*Edwards, H.; Lefmann, K.;* Lake, B.; *Nielsen, K.; Skaarup, P.*, Simulation of a complete inelastic neutron scattering experiment. *Appl. Phys. A - Mat. Sci. Process* (2002) v. 74 p. S1492-94

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*The Materials Research Department never hibernates.*

## abstract

Selected activities of the Materials Research Department at Risø National Laboratory during 2002 are described. The scientific work is described in five chapters and a survey is given of the Department's educational activities along with a list of published work, patents, prizes, organized meetings, and membership of committees. Furthermore, the main figures outlining the funding and expenditures of the Department are given and a list of staff members is included.

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| Inside cover: | The white poplar avenue at Risø |

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